

Opportunities for X-ray Absorption Spectroscopy at NSLS-II

Bruce Ravel

Synchrotron Methods Group
Ceramics Division
National Institute of Standards and Technology

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Intro

Infrastructure

Experiments

NSI S-II

My mandate from Tony

- ▶ New scientific directions and technical innovations in molecular environmental science and geochemistry XAS studies
- ▶ Instrumentation, optics, and computing trends
- ▶ New technologies and scientific possibilities for XAS at low concentration and small spot size

Fortunately he's given me 20 whole minutes to talk!

NSLS-II Sources and their XAS applications

Opportunities for
X-ray Absorption
Spectroscopy at
NSLS-II

Bruce Ravel

Intro

Infrastructure

Experiments

NSLS-II

Damping wiggler Highest flux for low concentrations, low detector efficiency, or pump-probe dynamics. $\sim 1 \mu$ microprobe. Resolution refining monochromator. Somewhat quick XAS. 5.5–90 keV.

DW side station Very high flux, unfocused tender x-rays
(*X15b/X19a on steroids*)

3 pole wiggler Conventional XAS (4–25 keV) (*X11a*). Quick XAS (*X18b*). $< 5 \mu$ microprobe (*X26a*).

Soft bend Low energy (100–1000 eV) XAS (*U7a*). Medium energy (1–6 keV) x-ray bulk XAS (*X15b/X19a*). Medium energy (1–6 keV) x-ray microprobe.

Undulator Smallest spot size with excellent flux, XAS by scanning the gap.

Hutch instrumentation

Essential to the E&ES community

- ▶ In-line controlled atmosphere chamber
- ▶ High-volume, high-throughput cryostats

Other things to consider

- ▶ Automated gas handling: ion chambers and/or hazardous gases
- ▶ Liquid delivery: water, LN
- ▶ Ample power and signal capacity
- ▶ Laboratory support

Detector technology

- ▶ Energy discriminating (Ge or Si-drift) will be the work-horse
- ▶ Wavelength dispersive (multilayer analyzers, bent Laue, etc) will fill many important niches
- ▶ High-rate and large number detectors from the instrumentation division

Data acquisition and data analysis software

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X-ray Absorption
Spectroscopy at
NSLS-II

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Intro

Infrastructure

Experiments

NSLS-II

Common DAQ infrastructure Data acquisition software should be as similar as possible on all beamlines. The DAQ solution should be **extensible**, **open source** and **platform agnostic**. A client/server framework (probably based on EPICS) is very attractive.

Beamline monitoring

Metadata

Analysis automation

Data acquisition and data analysis software

Opportunities for
X-ray Absorption
Spectroscopy at
NSLS-II

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Intro

Infrastructure

Experiments

NSLS-II

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Beamline monitoring Everything interesting about a beamline should be continuously monitored and archived. *Was the hutch door open at 3:07 am on March 3rd? What position was the sample stage at 27 minutes ago? What was the mono temperature when we observed all that noisy data?*

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Analysis automation High-quality data processing and analysis needs to be better incorporated into the DAQ architecture. Raw XAS data should be presented to the user with baseline processing. Automated fitting available for high-throughput or screening applications.

EXAFS at environmentally relevant concentrations

Opportunities for
X-ray Absorption
Spectroscopy at
NSLS-II

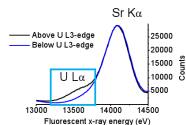
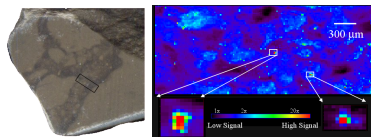
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Intro

Infrastructure

Experiments

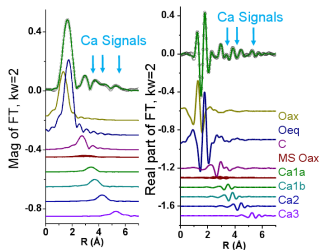
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Bulk: ~ 7 ppm U
Hot: ~ 18 ppm U

Evidence for stable uranium incorporation in an ancient calcite – a plausible sequestration strategy.

S.D. Kelly, E.T. Rasbury, S. Chattopadhyay, A.J. Kropf, K.M. Kemner, *Evidence of a stable uranyl site in ancient organic-rich calcite*. Environ. Sci. Technol. (2006), 40(7), 2262-2268.



After 2.5 days of continuous data collection at an APS undulator beamline, high quality data was obtained an subject to detailed analysis.

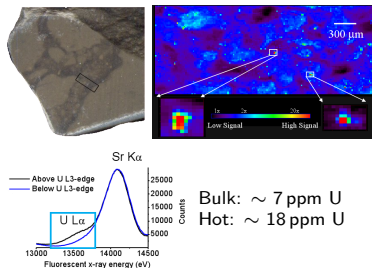
APS 10ID
NSLS X26a
NSLS-II DW

10^{11} ph/s 2.5 days

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Opportunities for
X-ray Absorption
Spectroscopy at
NSLS-II

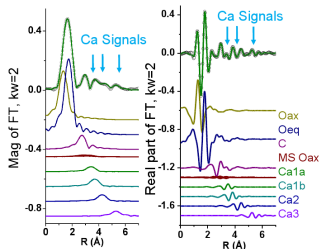
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Evidence for stable uranium incorporation in an ancient calcite – a plausible sequestration strategy.

2.5 days at 10^{11} ph/s \longrightarrow
 $\sim 2 \times 10^{16}$ total photons.

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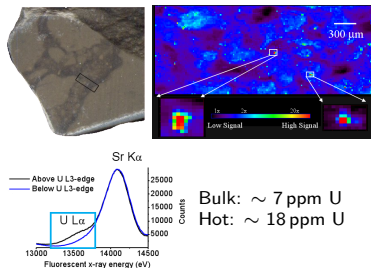
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Intro
 Infrastructure
 Experiments
 NSLS-II

EXAFS at environmentally relevant concentrations

Opportunities for
X-ray Absorption
Spectroscopy at
NSLS-II

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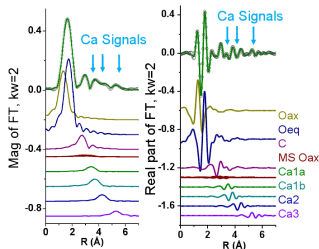


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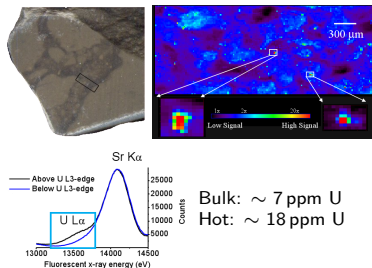
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NSLS X26a	3×10^8 ph/s	24 months
NSLS-II DW		

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Opportunities for
X-ray Absorption
Spectroscopy at
NSLS-II

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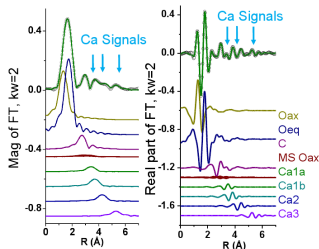


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NSLS X26a	3×10^8 ph/s	24 months
NSLS-II DW	10^{12} ph/s	6 hours

Intro
Infrastructure
Experiments
NSLS-II

High resolution fluorescence detection

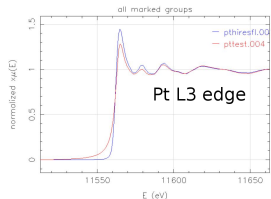
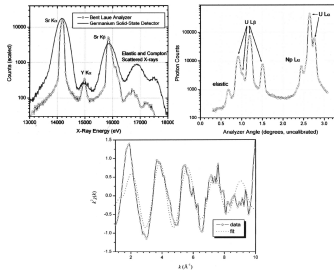
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X-ray Absorption
Spectroscopy at
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The bent Laue analyzer does wavelength dispersive detection of the fluorescence signal.

1 part Np in 160 parts U_3O_8



Imagine doing $\text{U}^{4+}/\text{U}^{6+}$
speciation with this level of
resolution!

A. J. Kropf, R. J. Finch, J. A. Fortner, S. Aase, C. Karanfil, C. U. Segre, J. Terry, G. Bunker, and L. D. Chapman, *Bent silicon crystal in the Laue geometry to resolve x-ray fluorescence for x-ray absorption spectroscopy*, Rev. Sci. Instrum. (2003), 74, 4696-4702

High-resolution EXAFS

With high flux, experiments
with low detector efficiency
are feasible.

Intro

Infrastructure

Experiments

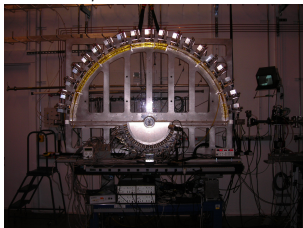
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LERIX: Soft XAS edges using hard x-rays

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X-ray Absorption
Spectroscopy at
NSLS-II

Bruce Ravel

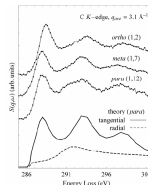
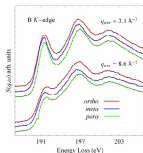
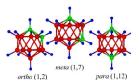
APS, Beamline 20ID



Incident energy: 10 keV

Near dipole conditions: 1750
and 320 cps at B and C K-edges
on top of 2360 and 1250 cps

Three isomers of $C_2B_{10}H_{12}$



T.T. Fister, F.D. Vila, G.T. Seidler, L. Svec,
J.C. Linehan, J.O. Cross, *The Local Electronic Structure
of Dicarba-closo-dodecarboranes $C_2B_{10}H_{12}$* , J. Am.
Chem. Soc. accepted

Too cool!

No vacuum, no special sample prep, hard x-rays in!

Intro

Infrastructure

Experiments

NSLS-II

What *you* should be thinking about

- ▶ What novel experiments can **you** imagine which use the new sources NSLS-II will provide?
- ▶ What instrumentation or infrastructure would **you** like to see at beamlines at NSLS-II?

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A special note to the university faculty among us

NSLS-II (and other synchrotrons) need good staff scientists. Encourage some of your students to fall in love with synchrotron radiation.